

Reference 22



*Protecting Texas
by Reducing and
Preventing Pollution*

Quality Assurance Project Plan

for

**Texas Natural Resource Conservation Commission
Preliminary Assessment/Site Inspection Program**

(FY 2000-2001)



October 1999

Quality Assurance Project Plan

Texas Natural Resource Conservation Commission
Preliminary Assessment/Site Inspection
Program (FY 2000-2001)

Prepared in cooperation with the
U.S. Environmental Protection Agency

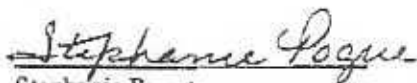
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QUALITY ASSURANCE PROJECT PLAN
FOR
TNRCC PRELIMINARY ASSESSMENT AND SCREENING SITE INSPECTION
PROGRAM

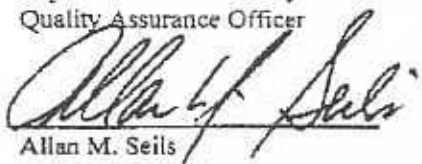
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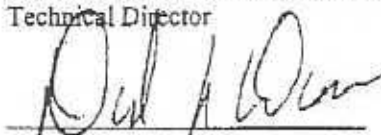
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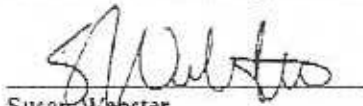
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Quick Reference Fact Sheet, August 1999

Appendix E - Low Concentration Organic Analytical Service for Superfund (Water Matrix)
Quick Reference Fact Sheet, August 1999

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1994.

SECTION B

MEASUREMENT/DATA ACQUISITION

(B1) SAMPLING PROCESS DESIGN

After approval of the SSI work plan, the field activities will be executed. At each site, these activities may include soil sampling, sediment sampling, surface water sampling, and groundwater sampling.

Detailed reports on all PA and SSI non-sampling data collection and SSI sampling activities will be kept in field logbooks. In this book will be noted the date, time, location, and identification of each sample, along with the collector's name, a description of all equipment used and any problems encountered, and general comments of the inspection team. Logbooks also are used to record pertinent information regarding the site itself, including date, time, location, and identification of all photographs taken during the site visit.

Proper identification and labeling of samples is crucial to an effective sampling program. Immediately upon collection, each sample must be sealed and tagged. The tag should be marked with a sample identification number, station location, type (composite or grab), concentration (low, medium, or high), the parameters requested, collector's name, and the date and time of sample collection.

For many of the SSIs, the determining factor of hazard evaluation will be the data provided by sampling and analytical activities. Thus, it is important that QA/QC be maintained for each sample. The purpose of this Section is to outline specific procedures for inspectors to use while acquiring and handling samples during an inspection to ensure that quality data are obtained.

Certified clean sample bottles will be used for sample collection. Custody of these bottles will be maintained by documenting the batch number of the sealed box, documenting opening of the box, and keeping the bottles locked up at all times. If returned to the office, the bottles will be placed in a sealable container and secured with custody seals.

(B2) SAMPLE METHODS REQUIREMENTS

This Section discusses the standard sampling procedures. Other sampling procedures may be used as determined necessary by the lead Site Investigation Manager and with approval of the Technical Director or Program Manager.

Regardless of sample type, the following principles and procedures should be adhered to during the sample collection phase of a site inspection:

1. Obtain ice before visiting a site where sample collection is involved.
2. Add appropriate preservatives to the sample bottles at the time of sample collection. See Appendix B, p. 14. The bottles required for each analysis are shown in Appendix B, p. 9-10.
3. If there is reason to suspect the presence of toxic vapors, precede sampling activities by an initial survey of suspect areas, using appropriate safety gear and a photoionization detector (or equivalent). The potential use of air monitoring equipment should have been specified in the SSI Work Plan. If it was not, and if organic vapor presence is possible, contact the Program Manager and Project Safety Officer for possible changes in safety procedures.
4. If possible, collect background samples first, then proceed from the probable least contaminated to most contaminated sampling points.
5. Change disposable gloves between sampling points, placing used gloves in a plastic bag for disposal.
6. If it is necessary to reuse sampling devices, use the specified decontamination procedures between sampling points.
7. At each sampling location,
 - a. Photograph the collection of samples.
 - b. Record in the logbook:
 - Sample number;
 - Photo number;
 - Location (show on site sketch);
 - Type of sample;
 - Date and Time; and
 - Relevant observations.

8. If a facility representative requests, they will be allowed the opportunity to collect split samples. If these are desired, place samples directly in different containers at the sampling point rather than splitting them at a later time. In the event there may not be enough soil, sediment, and/or groundwater volume to provide split samples, collect the SSI required sample first and then provide the remaining volume to the facility representative.
9. Before placing samples in the iced cooler:
 - a. Complete the sample tags and labels, and place clear tape over the sample labels on the sample containers to protect the writing from moisture.
 - b. Check the pH of all preserved water samples (exclusive of VOA samples).
 - c. Place a custody seal around the bottle cap.
 - d. Wrap the sample containers with plastic foam, bubble pack, or equivalent to protect against breakage.
 - e. The TNRCC will include in each ice chest with samples to be shipped for analysis, a temperature blank taped to the side of the chest prior to shipping.
 - f. Place the sample containers in plastic Ziploc® bags or equivalent to prevent melted ice from contacting the container.
 - g. Place wrapped sample containers into ice chests filled with 2 to 3 inches of vermiculite.
10. Remove water from melted ice frequently, and replace with fresh ice. Place ice in plastic Ziploc® or sealable bags to minimize water leakage during shipment.

SURFACE SOIL SAMPLING PROCEDURES

Areas selected for sampling will be located in order to collect a representative fraction of the soils with the minimum of samples. A surface inspection of the subject area will be made to locate pertinent features (e.g., rock outcrops, drainage patterns, surface runoff, erosion areas, etc.) and to evaluate the relationship among these features and potential sources of pollution. The locations of sediment deposition areas are good indicators of surface runoff direction.

A method of obtaining a shallow soil sample is to use stainless steel spoon or shovel. When deemed appropriate, a deeper soil sample may be obtained through the use of a soil corer. After collection, the soil sample will then be placed in the appropriate glass bottle. After the sample has been collected, the top of the bottle and lid will be wiped with a clean paper towel to ensure a tight seal. Samples for VOA analysis will be collected first, followed by samples for BNA's, metals and pesticides/PCBs. If metals are the primary concern at a site, the metals sample will be collected second. Care will be taken to fill the 120 mL VOA sample as full as possible to eliminate headspace. A decontaminated shovel or spade can be used to uncover the top 6 inches of soil so the sample can be collected from beneath the surface.

Sampling equipment such as stainless steel scoops and spoons, and shovels or spades must be decontaminated according to the specified procedures between sampling locations to avoid cross contamination. Dedicated sampling equipment will normally be used. If dedicated equipment are not used, then an equipment rinsate sample shall be collected at the end of each sampling day to demonstrate decontamination efficiency by TNRCC field personnel.

SEDIMENT SAMPLING PROCEDURES

Areas selected for sampling will be located in order to collect a representative fraction of the sediments with the minimum of samples. The primary consideration in sample site selection will be to choose an area of quiescent settling with low hydrologic activity or energy, and to evaluate these areas and potential sources of pollution. For example, areas that are: 1) inside the bend of channels; 2) backwater areas or side channels; and 3) of heavy shoaling and deposition. Quiescent areas are conducive to the settling of finer materials.

Sediment samples will be collected by use of a stainless steel spoon; or for samples greater than six (6) inches beneath the water surface, samples will be collected using either an Ekman dredge or sediment corer. When using a dredge, it will be lowered to the bottom of the water body with a minimum of substrate disturbance. Once the dredge jaws have been triggered, the closed dredge will be retrieved at a moderate speed of less than two (2) feet/second. Water overlying the sediment in the dredge will be gently decanted by slightly tipping the dredge until the water runs out the top. The decanting process will be completed in a manner to avoid

the removal of surficial sediments. In order to avoid contamination from material on the dredge walls, a stainless steel spoon will be used to remove sediments to a depth of one inch and no closer than 0.75 inches to the wall of the dredge. The sediment sample will then be placed in the appropriate glass bottle. Pebbles and sticks will not be transferred to the sample bottle. Additional dredge samples will be collected as needed to fill the sample bottle. After the sample has been collected, the top of the bottle and lid will be wiped with a clean paper towel to ensure a tight seal. Samples for VOA analysis will be collected first, followed by samples for BNA's, metals and pesticides/PCBs.

If metals are the primary concern at a site, the metals sample will be collected second. Care will be taken to fill the 120 mL VOA sample as full as possible to eliminate headspace. The Ekman dredge and stainless steel spoons must be decontaminated according to the specified procedures between sampling locations to avoid cross contamination. Dedicated sampling equipment will normally be used.

DECONTAMINATION PROCEDURES

To prevent contamination of samples by materials originating from the variety of on-site sampling tools and equipment, all sampling equipment (sample scoops, bailers, surface water dippers) will be decontaminated. Dedicated sampling equipment will be available for each sample planned. All equipment to be used at one site will be decontaminated in one batch prior to initiating any sampling. Each sampling tool will be placed in an individual sealable plastic bag or wrapped in a large plastic trash bag and closed with a custody seal. In the event that additional sampling is required or a sampling tool's integrity is questionable, then that tool will go through a decontamination process. The decontamination procedures are as follows:

1. Rinse equipment with tap (potable) water.
2. Clean the equipment with a brush in a solution of laboratory-grade detergent (Liquinox, Alconox, or equivalent) and potable water.
3. Rinse with tap water.
4. Rinse with 10 percent nitric acid solution, (trace metals grade) if analyzing for metals.

(B5) QUALITY CONTROL REQUIREMENTS

Quality assurance for analytical work on this project will involve analysis of blank samples, spiked samples, and duplicate samples. For each group of 20 samples (or less if fewer than 20 samples are collected) of similar matrix (i.e., groundwater/surface water, soil/sediment) collected at each site, CLP internal laboratory QA/QC analysis will be conducted on one blank, one spiked, and one duplicate spiked sample. Field duplicates will be collected at a rate of 10% for each matrix and/or one per day, whichever is greater. Also, the TNRCC will include in each ice chest with samples to be shipped for analysis a temperature blank taped to the side of the chest prior to shipping.

LABORATORY QUALITY CONTROL BLANKS, SPIKED BLANKS, AND MATRIX SPIKES

Analysis of blank samples verifies that the analytical method does not introduce contaminants. The spiked blank is generated by addition of standard solutions to the blank water. The matrix spike will be generated by the CLP laboratory through the addition of standard solutions to a randomly selected field sample. Extra volume (triple volume) for a matrix spike and matrix spike duplicate will be collected for one water sample (groundwater or surface water, but not both) by the field team and sent to the assigned CLP Laboratory for internal quality control. In addition, one soil sample (no extra volume) will be designated on the TR by the field team and sent to the designated CLP laboratory for internal quality control.

FIELD QUALITY CONTROL SAMPLES

All samples will be collected with dedicated equipment, if possible. All sampling equipment will be decontaminated prior to initiating sampling activities. Three types of blanks will be taken in the field. The first type, **field blanks**, are blanks that are exposed to the same contamination as the field samples (e.g., airborne contaminants that are not from the waste being sampled). The second type, **trip blanks**, are collected for volatiles only. Volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the Teflon-lined septum of the sample vial; therefore, a VOA **trip blank** will be analyzed to monitor for possible sample contamination. The trip blank also serves to detect contaminants in the sample bottles. These blanks are similar to field blanks with the exception that they are not exposed to field conditions. They allow evaluation of contamination generated from sample containers and changes occurring during the shipping and laboratory storage process. The third type, **equipment rinsate blanks**, will consist of CLP-specified grade water that has been poured over the equipment after completion of decontamination. The number of blanks collected in the field will be specified by the work plans for each site. The blanks collected in the field will not be counted for the laboratory's quality control protocol for matrix spikes or duplicate samples.

FIELD DUPLICATES

For samples collected for laboratory analysis, field duplicates will be collected at a rate of 10 percent of the total number of samples collected during each day of sampling for each sample matrix type at every site. The number of samples collected will be rounded up to the next increment of ten, such that twenty-one samples would require collection of three duplicates, if collected within three days. At least one field duplicate will be collected per day of sampling and will be packaged and sent to the laboratory for analysis with the other samples of the same sample matrix type.

EQUIPMENT RINSATE SAMPLES

Equipment rinsate samples will be collected to establish that proper sample bottle preparation, decontamination and handling techniques have been employed. Dedicated sample equipment will be used at each site for each sample station. All sample equipment will be decontaminated in the field and carefully packaged for return to the TNRCC Central Office. The decontaminated equipment will be taken to the TNRCC Region 11 Austin Office laboratory where one equipment blank will be collected and shipped to the assigned CLP laboratory for analysis. The equipment rinsate sample will be prepared by collecting CLP-specified grade water from the final rinse of the sampling equipment. Finally, the sample equipment will be placed in individual dated plastic bags, including chain-of-custody seals.

If sample equipment must be used more than once in the field, then the decontamination procedures for sample equipment will be followed and a rinsate sample collected in the field at the end of each sampling day and/or between each sample matrix type sampled, whichever is greater, and shipped to the assigned CLP laboratory with the associated sample matrix type. The number and type of QA samples at each site will be estimated in the SSI work plan. Modifications to the plan may be deemed necessary by the site investigation manager depending on field conditions, the on-site determination of additions or removals of sample locations, and the number of days required to complete the site sampling investigation.

(B7) CALIBRATION PROCEDURES AND FREQUENCY

Calibration of field instruments and equipment will be performed at approved intervals as specified by the manufacturer or more frequently as conditions dictate. Calibrations also may be performed at the start and completion of each test run. However, such calibrations will be re-initiated after any delay caused by meals, work shift change, or damage incurred. Standards will be used and duplicate samples analyzed in the field to verify pH and specific conductance data.

APPENDIX C

Multi-Media, Multi-Concentration, Organic Analytical Service
for Superfund

Quick Reference Fact Sheet, August 1999



Multi-Media, Multi-Concentration, Organic Analytical Service for Superfund (OLM03.2)

Office of Emergency and Remedial Response
Analytical Operations/Data Quality Center (5204G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to the EPA at such sites, are a series of volatile, semivolatile, and pesticide/Aroclor (pesticide/PCB) compounds that are analyzed using gas chromatography coupled with mass spectrometry (GC/MS) and gas chromatography with an electron capture detector (GC/ECD). The Analytical Operations/Data Quality Center (AOC) of the Office of Emergency and Remedial Response (OERR) offers an analytical service that provides data from the analysis of water and soil/sediment samples for organic compounds for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the organic analytical service produces data of known and documented quality. This service is available through the Superfund Contract Laboratory Program (CLP).

DESCRIPTION OF SERVICES

The organic analytical service provides a technical and contractual framework for laboratories to apply EPA/CLP analytical methods for the isolation, detection, and quantitative measurement of 33 volatile, 64 semivolatile, and 28 pesticide/Aroclor (pesticide/PCB) target compounds in water and soil/sediment environmental samples. The CLP provides the methods to be used and the specific technical, reporting, and contractual requirements, including quality assurance, quality control, and standard operating procedures, by which EPA evaluates the data. This service uses GC/MS and GC/ECD methods to analyze the target compounds. Two data delivery turnarounds are available to CLP customers: 35-day turnaround and 14-day turnaround after laboratory receipt of the last sample in the set.

DATA USES

This analytical service provides data which EPA uses for a variety of purposes, such as determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the

investigation of a hazardous waste site including site inspections, Hazard Ranking System scoring, remedial investigations/feasibility studies, remedial design, treatability studies, and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/litigation activities.

TARGET COMPOUNDS

The compounds for which this service is applicable and the corresponding quantitation limits are listed in Table 1. For water samples, the lowest quantitation limits reportable are 10 ppb for the volatile compounds, 10 ppb for the semivolatile compounds, and 0.05 ppb for the pesticide/Aroclor (pesticide/PCB) compounds. For soil samples, the lowest quantitation limits reportable are 10 ppb for the volatile compounds, 330 ppb for the semivolatile compounds, and 1.7 ppb for the pesticide/Aroclor (pesticide/PCB) compounds. Specific sample quantitation limits are highly matrix dependent. Compounds identified with concentrations below the quantitation limit are reported as estimated concentration values.

Table 1. Target Compound List and Contract Required Quantitation Limits (CROLs) For OLM03.2*

Quantitation Limits			Quantitation Limits			Quantitation Limits				
	Water (ug/L)	Low Soil (ug/Kg)		Water (ug/L)	Low Soil (ug/Kg)		Water (ug/L)	Low Soil (ug/Kg)		
<u>VOLATILES</u>			43.	N-Nitroso-di-n-propylamine	10	330	89.	Chrysene	10	330
1.	Chloromethane	10	44.	Hexachloroethane	10	330	90.	bis-(2-Ethylhexyl)phthalate	10	330
2.	Vinyl Chloride	10	45.	Nitrobenzene	10	330	91.	Di-n-octylphthalate	10	330
3.	Bromomethane	10	46.	Isophorone	10	330	92.	Benzo(b)fluoranthene	10	330
4.	Chloroethane	10	47.	2-Nitrophenol	10	330	93.	Benzo(k)fluoranthene	10	330
5.	1,1-Dichloroethene	10	48.	2,4-Dimethylphenol	10	330	94.	Benzo(a)pyrene	10	330
6.	Acetone	10	49.	bis-(2-Chloroethoxy)methane	10	330	95.	Indeno(1,2,3-cd)pyrene	10	330
7.	Carbon Disulfide	10	50.	2,4-Dichlorophenol	10	330	96.	Dibenzo(a,h)anthracene	10	330
8.	Methylene Chloride	10	51.	1,2,4-Trichlorobenzene	10	330	97.	Benzo(g,h,i)perylene	10	330
9.	1,2-Dichloroethene (total)	10	52.	Naphthalene	10	330	<u>PESTICIDES/AROCLORS</u>			
10.	1,1-Dichloroethane	10	53.	4-Chloroaniline	10	330	<u>(PESTICIDES/PCBs)</u>			
11.	2-Butanone	10	54.	Hexachlorobutadiene	10	330		Water	Soil	
12.	Chloroform	10	55.	4-Chloro-3-methylphenol	10	330		(ug/L)	(ug/Kg)	
13.	1,1,1-Trichloroethane	10	56.	2-Methylnaphthalene	10	330	98.	alpha-BHC	0.05	1.7
14.	Carbon Tetrachloride	10	57.	Hexachlorocyclopentadiene	10	330	99.	beta-BHC	0.05	1.7
15.	Benzene	10	58.	2,4,6-Trichlorophenol	10	330	100.	delta-BHC	0.05	1.7
16.	1,2-Dichloroethane	10	59.	2,4,5-Trichlorophenol	25	830	101.	gamma-BHC (Lindane)	0.05	1.7
17.	Trichloroethene	10	60.	2-Chloronaphthalene	10	330	102.	Heptachlor	0.05	1.7
18.	1,2-Dichloropropane	10	61.	2-Nitroaniline	25	830	103.	Aldrin	0.05	1.7
19.	Bromodichloromethane	10	62.	Dimethylphthalate	10	330	104.	Heptachlor epoxide	0.05	1.7
20.	cis-1,3-Dichloropropene	10	63.	Acenaphthylene	10	330	105.	Endosulfan I	0.05	1.7
21.	4-Methyl-2-pentanone	10	64.	2,6-Dinitrotoluene	10	330	106.	Dieldrin	0.10	3.3
22.	Toluene	10	65.	3-Nitroaniline	25	830	107.	4,4'-DDE	0.10	3.3
23.	trans-1,3-Dichloropropene	10	66.	Acenaphthene	10	330	108.	Endrin	0.10	3.3
24.	1,1,2-Trichloroethane	10	67.	2,4-Dinitrophenol	25	830	109.	Endosulfan II	0.10	3.3
25.	Tetrachloroethene	10	68.	4-Nitrophenol	25	830	110.	4,4'-DOD	0.10	3.3
26.	2-Hexanone	10	69.	Dibenzofuran	10	330	111.	Endosulfan sulfate	0.10	3.3
27.	Dibromochloromethane	10	70.	2,4-Dinitrotoluene	10	330	112.	4,4'-DDT	0.10	3.3
28.	Chlorobenzene	10	71.	Fluorene	10	330	113.	Methoxychlor	0.50	17
29.	Ethylbenzene	10	72.	Diethylphthalate	10	330	114.	Endrin ketone	0.10	3.3
30.	Xylenes (total)	10	73.	4-Chlorophenyl-phenylether	10	330	115.	Endrin aldehyde	0.10	3.3
31.	Styrene	10	74.	4-Nitroaniline	25	830	116.	alpha-Chlordane	0.05	1.7
32.	Bromoform	10	75.	4,6-Dinitro-2-methylphenol	25	830	117.	gamma-Chlordane	0.05	1.7
33.	1,1,2,2-Tetrachloroethane	10	76.	N-Nitrosodiphenylamine	10	330	118.	Toxaphene	5.0	170
			77.	4-Bromophenyl-phenylether	10	330	119.	Aroclor-1016	1.0	33
			78.	Hexachlorobenzene	10	330	120.	Aroclor-1221	2.0	67
			79.	Pentachlorophenol	25	830	121.	Aroclor-1232	1.0	33
			80.	Phenanthrene	10	330	122.	Aroclor-1242	1.0	33
			81.	Anthracene	10	330	123.	Aroclor-1248	1.0	33
			82.	Carbazole	10	330	124.	Aroclor-1254	1.0	33
			83.	Di-n-butylphthalate	10	330	125.	Aroclor-1260	1.0	33
			84.	Fluoranthene	10	330				
			85.	Pyrene	10	330				
			86.	Burylbenzylphthalate	10	330				
			87.	3,3'-Dichlorobenzidine	10	330				
			88.	Benzo(a)anthracene	10	330				
<u>SEMIVOLATILES</u>										
34.	Phenol	10	330							
35.	bis-(2-Chloroethyl)ether	10	330							
36.	2-Chlorophenol	10	330							
37.	1,3-Dichlorobenzene	10	330							
38.	1,4-Dichlorobenzene	10	330							
39.	1,2-Dichlorobenzene	10	330							
40.	2-Methylphenol	10	330							
41.	2,2'-oxybis (1-Chloropropane)	10	330							
42.	4-Methylphenol	10	330							

* For volatiles, quantitation limits for medium soils are approximately 120 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 30 times the quantitation limits for low soils.

The list of target compounds for this service was originally derived from the EPA Priority Pollutant List of 129 compounds. In the years since the inception of the CLP, compounds have been added to and deleted from the Target Compound List (TCL), based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program.

For drinking water and groundwater type samples, use of the low concentration organic analytical service is recommended.

METHODS AND INSTRUMENTATION

For semivolatile and pesticide/Aroclor (pesticide/PCB) water samples, a 1-L aliquot is extracted with methylene chloride using a continuous liquid-liquid extractor or separatory funnel (for pesticides/Aroclors (pesticides/PCBs) only). For low level semivolatile soil and pesticide/Aroclor (pesticide/PCB) soil samples, a 30-g soil sample is extracted with methylene chloride/acetone. For medium level semivolatile soil samples, a 1-g soil sample is extracted with methylene chloride using sonication. For both water and soil samples, the extract is concentrated, subjected to fraction-specific cleanup procedures, and analyzed by GC/MS for semivolatiles or GC/ECD for pesticides/Aroclors (pesticides/PCBs).

For volatile water samples, 5 mL of water is added to a purge and trap device and purged with an inert gas at room temperature. For volatile low level soil samples, a 5-g aliquot of soil is added to a purge and trap device with 5 mL of reagent water and purged with an inert gas at 40°C. For volatile medium level soil samples, 4 g are extracted with methanol and an aliquot is added to reagent water. For both water and soil samples, the volatiles purged from the sample are trapped on a solid sorbent. They are subsequently desorbed by rapidly heating the sorbent and then introduced into a GC/MS system. Table 2 summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for this service include both hardcopy/electronic data reporting forms and supporting raw data. The laboratory must submit data to EPA within 35 days (or 14 days for 14-day contracts) after laboratory receipt of the last sample in the set. EPA then processes the data through an automated Data Assessment Tool. DAT is a complete CLP data assessment package. DAT incorporates Contract Compliance Screening (CCS) and Computer-Aided Data Review and Evaluation (CADRE) review to provide EPA Regions with PC-compatible reports, spreadsheets, and electronic files within 24 to 48 hours from the receipt of the data for data validation. This automated tool facilitates the transfer of analytical data into Regional databases. In addition to the Regional electronic

reports, the CLP laboratories are provided with a data assessment report that documents the instances of noncompliance. The laboratory has 10 days to reconcile defective data and resubmit the data to EPA. EPA then reviews the data for noncompliance and sends a final data assessment report to the CLP laboratory and the Region.

QUALITY ASSURANCE

The quality assurance (QA) process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity. This process ensures that the data provided are of the quality required. During the implementation of the data collection effort, QA activities ensure that the quality control (QC) system is functioning effectively and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data to determine its suitability to support enforcement or remedial decisions.

Each contract laboratory prepares a quality assurance plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, and functional guidelines, as well as the QA and QC activities designed to achieve the data quality requirements for this analytical service.

QUALITY CONTROL

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for or the effect of corrective action procedures. The QC procedures required for this analytical service are shown in Table 3.

Table 2. Methods and Instruments

Fraction	Water	Soil
Volatiles	Purge and trap followed by GC/MS analysis	Purge and trap followed by GC/MS analysis
Semivolatiles	Continuous liquid-liquid extraction followed by GC/MS analysis	Sonication followed by GC/MS analysis
Pesticides/Aroclors (Pesticides/PCBs)	Continuous liquid-liquid or separatory funnel extraction followed by dual column GC/ECD analysis	Sonication followed by dual column GC/ECD analysis

Table 3. Quality Control

QC Operation	Frequency
System Monitoring Compounds (volatiles)	Added to each sample, standard, and blank
Surrogates (for semivolatiles and pesticides/Aroclors (pesticides/PCBs))	Added to each sample, standard, and blank
Method Blanks (volatiles)	Analyzed at least every 12 hours for each matrix and level
Method Blanks (semivolatiles and pesticides/Aroclors (pesticides/PCBs))	Prepared with each group of 20 samples or less of the same matrix and level, or each time samples are extracted
Instrument Blank (volatiles)	Analyzed after a sample which contains compounds at concentrations greater than the calibration range
Instrument Blank (pesticides/Aroclors (pesticides/PCBs))	Every 12 hours on each GC column used for analysis
Storage Blanks (volatiles)	Prepared and stored with each set of samples
GC/MS mass calibration and ion abundance patterns (volatiles and semivolatiles)	Every 12 hours for each instrument used for analysis
GC Resolution Check (pesticides/Aroclors (pesticides/PCBs))	Prior to initial calibration, on each instrument used for analysis
Initial Calibration	Upon initial set up of each instrument, and each time continuing calibration fails to meet the acceptance criteria
Continuing Calibration	Every 12 hours for each instrument used for analysis
Internal Standards (volatiles and semivolatiles)	Added to each sample, standard, and blank
Matrix Spike and Matrix Spike Duplicate	Once every 20 or fewer samples of same fraction, matrix, and level

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by AOC and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, and GC/MS tape audits, and evaluates laboratory performance through the use of blind performance evaluation samples.

For more information, or for suggestions to improve this analytical service, please contact:

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APPENDIX D

Multi-Media, Multi-Concentration, Inorganic Analytical Service
for Superfund

Quick Reference Fact Sheet, August 1999



Multi-Media, Multi-Concentration, Inorganic Analytical Service for Superfund (ILM04.0)

Office of Emergency and Remedial Response
Analytical Operations/Data Quality Center (5204G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to the EPA at such sites, are a series of inorganic analytes and cyanide that are analyzed using inductively coupled plasma (ICP), atomic absorption (AA), and colorimetric techniques. The Analytical Operations/Data Quality Center (AOC) of the Office of Emergency and Remedial Response (OERR) offers an analytical service that provides data from the analysis of water and soil/sediment samples for inorganic analytes for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the inorganic analytical service produces data of known and documented quality. This service is available through the Superfund Contract Laboratory Program (CLP).

DESCRIPTION OF SERVICES

The inorganic analytical service provides a technical and contractual framework for laboratories to apply EPA/CLP analytical methods for the preparation, detection, and quantitative measurement of 23 inorganic target analytes and cyanide in water and soil/sediment environmental samples. The CLP provides the methods to be used and the specific technical, reporting, and contractual requirements, including quality assurance, quality control, and standard operating procedures, by which EPA evaluates the data. This service uses inductively coupled plasma, atomic absorption, and colorimetric methods to analyze the inorganic target analytes and cyanide. Two data delivery turnarounds are available to CLP customers: 35-day turnaround and 14-day turnaround after laboratory receipt of the last sample in the set.

DATA USES

This analytical service provides data which EPA uses for a variety of purposes, such as determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions

are complete. The data may be used in all stages in the investigation of a hazardous waste site including site inspections, Hazard Ranking System scoring, remedial investigations/feasibility studies, remedial design, treatability studies, and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/litigation activities.

TARGET ANALYTES

The analytes and detection limits for which this service is applicable are listed in Table 1. The list of target analytes for this service was originally derived from the EPA Priority Pollutant List of 129 compounds. In the years since the inception of the CLP, analytes have been added to and deleted from the Target Analyte List, based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program. Specific detection limits are highly matrix dependent.

METHODS AND INSTRUMENTATION

Table 2 summarizes the methods and instruments used in this analytical service.

Table 1
Target Analyte List and Contract Required
Detection Limits (CRDLs) (ILM04.0)

Abbreviation	Analyte	Contract Required Detection Limit ¹ (ug/L)
Al	Aluminum	200
Sb	Antimony	60
As	Arsenic	10
Ba	Barium	200
Be	Beryllium	5
Cd	Cadmium	5
Ca	Calcium	5000
Cr	Chromium	10
Co	Cobalt	50
Cu	Copper	25
Fe	Iron	100
Pb	Lead	3
Mg	Magnesium	5000
Mn	Manganese	15
Hg	Mercury	0.2
Ni	Nickel	40
K	Potassium	5000
Se	Selenium	5
Ag	Silver	10
Na	Sodium	5000
Tl	Thallium	10
V	Vanadium	50
Zn	Zinc	20
Cn	Cyanide	10

Sample concentration exceeding five times the detection limit of the instrument or method in use may be reported even though the instrument or method detection limit is greater than the CRDL. This is illustrated in the following example:

For lead:

Method in use = ICP

Instrument Detection Limit (IDL) = 40

Sample Concentration = 220

Contract Required Detection Limit (CRDL) = 3

¹The Contract Required Detection Limit is the instrument detection limit obtained in pure water.

DATA DELIVERABLES

Data deliverables for this service include both hardcopy/electronic data reporting forms and supporting raw data. The laboratory must submit data to EPA within 35 days (or 14 days for 14-day contracts) after laboratory receipt of the last sample in the set. EPA then processes the data through an automated Data Assessment Tool. DAT is a complete CLP data assessment package. DAT incorporates Contract Compliance Screening (CCS) and Computer-Aided Data Review and Evaluation (CADRE) review to provide EPA Regions with PC-compatible reports, spreadsheets, and electronic files within 24 to 48 hours from the receipt of the data for data validation. This automated tool facilitates the transfer of analytical data into Regional databases. In addition to the Regional electronic reports, the CLP laboratories are provided with a data assessment report that documents the instances of noncompliance. The laboratory has 10 days to reconcile defective data and resubmit the data to EPA. EPA then reviews the data for noncompliance and sends a final data assessment report to the CLP laboratory and the Region.

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QUALITY CONTROL

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for or the effect of corrective action procedures. The QC procedures required for this analytical service are shown in Table 3.

Table 2. Methods and Instruments

Analyte	Instrument	Method
Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Ti, V, Zn	Inductively Coupled Plasma (ICP)	Acid digestion followed by ICP analysis
As, Pb, Ti, Se	Graphite Furnace Atomic Absorption (GFAA)	Acid digestion followed by GFAA analysis
Ca, Mg, Na, K	Flame Atomic Absorption (FAA)	Acid digestion followed by FAA analysis
Hg	Cold Vapor Atomic Absorption (CVAA)	Acid and permanganate oxidation followed by CVAA analysis
CN	Manual and Semi-automated Colorimetric	Distillation followed by colorimetric analysis

Table 3. Quality Control

QC Operation	Frequency
Instrument Calibration	Daily or each time instrument is set up
Initial Calibration Verification	Following each instrument calibration
Initial Calibration Blank	Following each instrument calibration
Continuing Calibration Verification	Every ten analytical samples or every two hours during a run and at the beginning and end of each run
Continuing Calibration Blank	Every ten analytical samples or every two hours during a run and at the beginning and end of each run
Interference Check Sample	Every twenty analytical samples and at the beginning and end of each run
CRDL Standard for ICP	Every twenty analytical samples and at the beginning and end of each run
CRDL Standard for AA	At the beginning of each AA analytical run
Serial Dilution	For each matrix type and concentration for each sample delivery group (SDG)
Preparation Blank	For each sample preparation, analysis, and matrix per batch of prepared samples
Laboratory Control Sample	For each sample preparation and analysis procedure for each batch
Matrix Spike Sample Analysis	For each matrix type, concentration level, and method for each SDG
Duplicate Sample Analysis	For each matrix type, concentration level, and method for each SDG
Post Digestion Spike	Each time matrix spike recovery is outside QC limits
Analytical Spike	For each analytical sample analyzed by furnace AA
Method of Standard Addition	When the analytical spike recovery is outside QC limits
Instrument Detection Limit Determination	Quarterly
Interelement Corrections	Annually for ICP instruments only
Linear Range Analysis	Quarterly for ICP instruments only

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by AOC and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, and evaluates laboratory performance through the use of blind performance evaluation samples.

For more information, or for suggestions to improve this analytical service, please contact:

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